

Monitoring Nitric Oxide Loading and Release Processes on Silicone Rubber Substrates using Diffuse Reflectance Spectroscopy

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N-diazoniumdiolates are an established class of nitric oxide (NO) donor that have been of interest for synthesizing materials with the capability to store and release NO for biomaterials applications. The kinetics of *N*-diazoniumdiolate formation and decomposition have been established for certain small molecule donors in solution phase, however these processes have not been investigated for a solid material substrate. In order to optimize the synthesis of NO-materials, donor formation characterization must be established for a variety of synthetic conditions. Further, to assess the therapeutic action of the material, the rate of NO release from *N*-diazoniumdiolated materials must be directly probed. Herein, we describe a diffuse reflectance spectroscopy technique that can probe the *N*-diazoniumdiolate moiety within a cured silicone rubber thin film. Prior to film curing, the silicone rubber is modified to contain different secondary amine sites that can house the *N*-diazoniumdiolate moiety. We demonstrate the rate of donor moiety formation under different conditions by monitoring the formation of the *N*-diazoniumdiolate absorption band. We alter the pressure and purity of the NO gas as well the temperature of the reaction cell to assess the impact of these external variables on donor moiety formation; internal material variables, such as the amine structure and composition of the silicone rubber film are also altered. Once the NO loading process is characterized, we then consider the rate of NO delivery from the material under thermal conditions by monitoring the decomposition of the *N*-diazoniumdiolate moiety absorption. Using a diffuse reflectance setup, we are able to monitor the kinetics of NO loading and release from a solid substrate directly for the first time. These studies indicate the effect of the matrix environment and reaction conditions on material processes that can be harnessed to fine-tune the synthesis of biomaterials that release the therapeutic agent NO.